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(Final Report)

ARMOUR RESEARCH FOUNDATION OF ILLINOIS INSTITUTE OF TECHNOLOGY

TECHNOLOGY CENTER

IMPROVED VANADIUM-BASE ALLOYS

Contract NOw 61-0417-c

for

Department of the Navy
Bureau of Naval Weapons
Washington 25, D. C.

Attention: Code RRMA-211

62-2-3

25 years of research

ARMOUR RESEARCH FOUNDATION
of
ILLINOIS INSTITUTE OF TECHNOLOGY
Technology Center
Chicago 16, Illinois

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ABSTRACT

Alloys based on V-5w/oTi-20w/oCb and V-60w/oCb were found to exhibit excellent fabricability and weldability with good retention of strength to at least 2200°F. Ultimate tensile strength values of materials at the 60w/o columbium level measured under helium were above 60,000 psi at 2000°F and up to 44,400 psi at 2200°F; the strongest alloy at 2200°F was V-1w/oHf-60w/oCb. Materials at the 20w/o columbium level were at least 30% lower in strength at all temperatures. Stress-rupture properties at 2000°F of compositions based on V-5w/oTi-20w/oCb and V-60w/oCb were substantially improved by addition of 0.05w/o carbon. Interstitial impurities in the raw materials and those picked up during melting and hot working were found to reduce workability and to increase elevated-temperature strength. Tensile data obtained at elevated temperatures under vacuum were lower than those measured under helium due to reduced pickup of interstitials during testing. Alloys based on V-Ti-(Cb)-Si exhibited interesting age-hardening characteristics at 1200°F. Exposure to a saline environment under stress at 800° and 1000°F did not impair the mechanical properties of V-5w/oTi-20w/oCb, although slight pitting was observed at 1000°F.

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IMPROVED VANADIUM-BASE ALLOYS

I. INTRODUCTION

This report summarizes the work performed during the period December 1, 1960, to November 30, 1961, under Contract NOw 61-0417-c, entitled "Improved Vanadium-Base Alloys". Experimental work reported herein is a continuation of studies conducted under Contracts NOas 60-6056-c and NOas 59-6050-c. Some data from the earlier programs are included for purposes of comparison and to present a more complete description of the status of vanadium alloy development.

Previous studies of vanadium-base compositions demonstrated the outstanding weldability and fabricability as well as the excellent high-temperature strength of V-Cb-Ti materials. During the current year, emphasis was placed on further complexing of V-Cb base alloys in an effort to improve the strength properties at 2000°F and above. In addition to these alloy development efforts, a program has been initiated under Contract NOw 61-0806-c which is concerned with the development of oxidation-protective coatings for these vanadium materials. Current results on this program indicate promise for silicide and other coatings--protection of V-1Ti-60Cb* has been afforded for 500 hours in 2000°F air with no evidence of coating failure. As a logical sequence of alloy development work, a pilot evaluation program has recently been initiated under Contract NOw 62-0101-c. Under this program, large ingots (150 pounds) of the most promising compositions will be fabricated to sheet for detailed property evaluations.

The vanadium alloys described in this report were prepared, with one exception, from 200-gram nonconsumable-electrode arc-melted ingots. Almost eighty compositions were studied in addition to the nearly two hundred alloys evaluated during the preceding programs. Most of the materials under study are based on the vanadium-columbium system and are intended for applications at temperatures of 2000°F and above; some age-hardenable alloys were also investigated for use at lower temperatures

* Compositions are reported in weight per cent.

(near 1200°F). New compositions were screened by tensile tests at room temperature and at temperatures up to 2200°F. Stress-rupture, creep, age-hardening and other elevated temperature properties were determined for the more promising materials. Fabricability, weldability, and salt-corrosion behavior were also investigated. The best combinations of fabricability and elevated-temperature strength have been found in alloys based on V-5Ti-20Cb and V-60Cb. At present, optimum quantities of the interstitials carbon, nitrogen, and oxygen for high-temperature strengthening of these alloys have not been determined. Consequently, this phase of the alloy development effort will be continued under the pilot evaluation program prior to selection of compositions for the large ingots.

II. EXPERIMENTAL METHODS

A. Materials

Vanadium, columbium, and titanium were the major constituents of the alloys studied under this program. Impurity analyses of these materials are shown in Table I. Minor alloying additions were made with commercially available melting stock of high purity. The vanadium stock used during the current year has lower carbon and oxygen levels, but a higher nitrogen content than melting stock used on preceding programs. As arc-melted, the vanadium has a hardness of 147 VPN, or 80 R_B, slightly lower than the hardness of previously used stock. Two grades of columbium were used; Table I shows that the electron-beam melted stock had higher interstitial levels than were reported for the Wah Chang sheet stock.

B. Melting

With the exception of a 50-pound ingot produced by a commercial source, all experimental alloys were prepared by nonconsumable-electrode arc-melting. These 200-gram ingots were melted five times, inverting after each melt, under an atmosphere of argon. Weight losses after melting were always less than 0. 1 gram, indicating that compositions were close to the desired nominal ranges. The 50-pound ingot of V-5Ti-20Cb was prepared by consumable-electrode arc-melting under vacuum at the Universal-Cyclops Corporation; this material was melted twice to insure good homogeneity.

TABLE I

MATERIALS FOR ALLOY PREPARATION

Element	Source	Impurity Analysis (w/o)[a]
Vanadium	Union Carbide Metal Co.	0.024C, 0.055O, 0.039N, 0.0019H
Columbium	Wah Chang Corp.	0.0030C, 0.0135O, 0 0098N, 0.04Ta
	Union Carbide Metals Co.[b]	0.008C, 0.04O, 0.01N, 0.0035H
Titanium	E. I. DuPont de Nemours	0.04Fe

a. Supplier's analyses

b. Electron-beam melted.

C. Sheet Fabrication

Fabrication of the 200-gram arc-melted ingots was accomplished either by hot rolling followed by cold rolling, or the as-cast ingots were cold-rolled directly to sheet. Prior to rolling, the ingots were machined on the top and bottom to remove any surface irregularities. Hot rolling was carried out in evacuated stainless steel cans at 2200° to 2300°F; after a reduction in thickness of about 30%, the steel casing was removed and the ingots were further reduced by cold rolling The final sheet thickness of 0.050 inch represents a total reduction in thickness of almost 90%.

Fabrication of the 6-inch diameter, 50-pound ingot of V-5Ti-20Cb involved the following steps: (1) after machining to a diameter of 5-1/4 inches and encapsulating in mold steel, the ingot was extruded to a 3-inch diameter at 2300°F using 54 to 75 tsi pressure; (2) the extruded billet, encapsulated in stainless steel, was hammer forged at 1500°F to a 1-3/4 inch thick bar; and (3) subsequent reduction to 0.050 inch thick was accomplished by cold-rolling. During the latter operation, the material was annealed at 2000°F after each 50% reduction in thickness.

D. Mechanical Property Evaluations

Vickers 10 kg hardness measurements were taken on polished metallographic specimens of alloys in sheet form. Microhardness values (25 to 100-gram loads) were also measured on the polished samples to investigate the extent of surface contamination, etc.

Sheet-test specimens for tensile and stress-rupture evaluations were machined from annealed, recrystallized 0.050 inch thick rolled stock. These specimens were 2-1/4 inches long and had gage sections 1/4 inch wide by 3/4 inch long.

Tensile data were obtained using a strain rate of 0.06 in/in/min. The large number of compositions under study were screened at elevated temperatures using a capsule which was rapidly heated and was continuously purged with a small flow of helium. Thermocouples were attached to each shoulder of the specimen and the capsule assembly was placed within a heated furnace. Approximately 30 to 45 minutes was required to attain temperatures in the 1800° to 2200°F range; the test temperature was maintained for 10 minutes prior to application of the load. A few specimens were evaluated at elevated temperatures under a vacuum of about 0.04 microns and also in a highly purified static helium atmosphere. Again, the 0.06 in/in/min strain rate was used.

Stress-rupture properties were evaluated at 2000°F using a pan-loaded 20:1 lever ratio machine. These tests were conducted in an Inconel capsule which contained a purified helium atmosphere; a slight positive pressure and a low flow rate of the gas were used.

E. Miscellaneous Evaluations

Many of the vanadium-base alloys under study were evaluated for bend ductility, workability, weldability, recrystallization temperature, age-hardening effects, and salt-corrosion resistance. The methods used to investigate these properties are described under subsequent sections of this report. In addition, sheet materials were supplied to other organizations for evaluation of specific properties. These results are also summarized in a subsequent section.

III. RESULTS AND DISCUSSION

Vanadium alloy development studies conducted during the preceding two-year period indicated that excellent fabricability and high-temperature strength were found in the V-5Ti-20Cb and V-60Cb compositions. A large number of alloys were investigated at the 20w/o columbium level, and 5w/o titanium produced maximum strengthening; small carbon additions to V-5Ti-20Cb improved the stress-rupture life at 2000°F. A pronounced peak in the elevated-temperature strength curve for V-Cb alloys was found at the 60w/o columbium level. At 2000°F, this composition had an ultimate tensile strength near 60,000 psi, considerably higher than any alloy previously evaluated. Excellent room-temperature ductility and fabricability were also noted for V-60Cb. Consequently, a large portion of alloy development efforts during the current year were devoted to optimizing compositions at this high columbium level. As previous studies had indicated promise for vanadium alloys for use at or near 1200°F, several age-hardenable compositions for this category were also evaluated.

A. Fabricability

The majority of materials prepared as 200-gram arc-melted ingots were readily fabricated to 0 050 inch sheet by either hot or cold rolling methods, and all compositions which were initially hot worked could be cold rolled the last 50 per cent reduction. Table II summarized the fabricability of the alloys studied during the current year. Only eight ingots could not be rendered to satisfactory 0.050 inch thick sheet. These consisted of V-5Ti-20Cb or V-1Ti-60Cb compositions containing 0.07w/o oxygen or nitrogen additions, and several alloys containing ternary or quaternary additions of chromium (1 to 15w/o), aluminum (10w/o), or tungsten (10w/o). (Compositions at lower aluminum and tungsten levels were successfully fabricated under this program or during the preceding year.) The majority of alloys could be cold-rolled directly from the cast and surface-machined ingots. Table II shows that fabricability was reduced by small additions of boron, carbon, oxygen, or nitrogen, and by 5w/o of molybdenum or tungsten.

The excellent fabricability of V-5Ti-20Cb was further substantiated by the ability to produce excellent 0.050 inch sheet from the 50-pound consumable-electrode arc-melted ingot. After extrusion to 3-inch diameter bar, the major portion of this material was hammer forged at 1500°F to 1 3/4 inch diameter bar followed by cold rolling (with intermediate 2000°F anneals) to sheet. Cylindrical sections of the extruded bar 3/8 inch thick were also cold-rolled directly to sheet.

Several compositions prepared during the current year had also been arc-melted and fabricated during the preceding alloy development programs using raw materials of lower purity. It was observed that melting stock containing lower total interstitial content exhibited improved fabricability. For example, V-5Ti-20Cb prepared under earlier programs required hot-working to break up the as-cast structure, whereas the more recent ingots prepared from higher-purity melting stock could be cold-rolled. A similar improvement in fabricability was noted for V-30Cb when prepared with high-purity vanadium. The effects of interstitials upon high-temperature strength properties are discussed in greater detail under a subsequent section of this report.

B. Annealing and Metallographic Studies

Recrystallization temperatures of alloys under study were determined by annealing cold-rolled sheet specimens for 1/2 hour at 2000°, 2200°, 2400°, and 2600° F. The samples, encapsulated in Vycor or quartz, were water quenched from the annealing temperature and evaluated by metallographic examination and hardness tests.

It was observed that the recrystallization behavior of alloys based on V-5Ti-20Cb was similar to that of materials based on V-1Ti-60 Cb. In both cases, oxygen and nitrogen did not raise the recrystallization temperature (2000° F) whereas carbon added to both bases produced a 200° F rise, and boron additions resulted in a 400° F increase in recrystallization temperature. Replacing the titanium with hafnium or zirconium did not alter the recrystallization behavior of the two V-Cb base compositions.

Hardness curves obtained on these materials revealed that, in general, the minimum hardness occurred after annealing 200° F below the recrystallization temperature; an exception was found for boron-containing alloys where the lowest hardness was found upon annealing 400° F below the recrystallization temperature. Annealing at higher temperatures caused increased solution of precipitates which produced higher hardness levels. Additions of 0.025w/o boron, carbon, nitrogen, or oxygen usually resulted in a 15 to 20 VPN increase above the hardness of the base alloy.

In addition to the solid-solution and dispersion-hardened alloys for use at temperatures of 2000° F or above, several age-hardenable alloys were evaluated for 1200° F application. This category includes eleven compositions containing 0.5, 1, or 2 w/o silicon, and also five alloys containing relatively large (10 to 25w/o) additions of aluminum or chromium. Titanium levels were generally 20w/o, and columbium levels ranged up to 40w/o (refer to Table II). Annealing studies revealed that all of these compositions were completely recrystallized at 2200° F. Complete solution of precipitates in the materials containing 0.5 and 1w/o silicon occurred after annealing 1/2 hour at 2400° F. Silicides were evident in the microstructures of the alloys containing 2w/o silicon at all annealing treatments including 2600° F; these materials exhibited high hardness values (above 350 VPN) after annealing.

TABLE II

VANADIUM ALLOYS PREPARED

Composition (w/o)	Working Process[a]	Composition (w/o)	Working Process[a]
20Cb	HR	60Cb	CR
0.2Ti-20Cb	HR	0.5Ti-60Cb	CR
2.5Ti-20Cb	HR	1Ti-60Cb	CR
2.5Ti-20Cb-0.25Hf	HR	2.5Ti-60Cb	CR
2.5Ti-20Cb-0.25Zr	HR	0.5Hf-60Cb	CR
2.5Ti-20Cb-0.5Hf	HR	1Hf-60Cb	CR
2.5Ti-20Cb-0.5Zr	HR	2Hf-60Cb	HR
5Ti-20Cb	CR	0.5Zr-60Cb	CR
5Ti-20Cb-0.25Hf	CR	1Zr-60Cb	CR
5Ti-20Cb-0.25Zr	CR	2Zr-60Cb	CR
5Ti-20Cb-0.5Hf	CR	1Ti-60Cb-3Mo	HR
5Ti-20Cb-0.5Zr	CR	1Ti-60Cb-5W	HR
5Ti-20Cb-0.03C	HR	1Ti-60Cb-10W	HR[b]
5Ti-20Cb-0.05C	HR	1Ti-60Cb-10Ta	CR
5Ti-20Cb-0.1C	HR	1Ti-60Cb-0.25C	HR
5Ti-20Cb-0.025B	HR	1Ti-60Cb-0.05C	HR
5Ti-20Cb-0.05B	HR	1Ti-60Cb-0.025O	HR
5Ti-20Cb-0.025N	HR	1Ti-60Cb-0.05O	HR
5Ti-20Cb-0.05N	HR	1Ti-60Cb-0.07O	HR[b]
5Ti-20Cb-0.025O	HR	1Ti-60Cb-0.025B	HR
5Ti-20Cb-0.05O	HR	1Ti-60Cb-0.05B	HR
5Ti-20Cb-0.07O	HR[b]	1Ti-60Cb-0.025N	HR
5Ti-20Cb-0.5Zr-0.03C	HR	1Ti-60Cb-0.05N	HR
5Ti-20Cb-0.5Zr-0.05	HR	1Ti-60Cb-0.025C-0.025B	HR
5Ti-20Cb-0.025C-0.025B	HR	1Hf-60Cb-0.025O	HR
5Ti-25Cb	CR	1Hf-60Cb-0.025C	HR
30Cb[c]	CR	1Hf-60Cb-0.05C	HR[b]
30Cb	CR[b]	1Hf-60Cb-5W	HR
5Ti-40Cb-0.03C	HR	85Cb	HR
4Hf-40Cb-0.1C	HR	90Cb	HR

TABLE II (continued)

Composition (w/o)	Working Process[a]	Composition (w/o)	Working Process[a]
	Alloys Primarily for 1200°F Application		
20Ti-0.5Si	HR	20Ti-40Cb-0.5Si	HR
20Ti-1.0Si	HR	20Ti-40Cb-1.0Si	HR
20Ti-2.0Si	HR	20Ti-40Cb-2.0Si	HR
10Ti-20Cb-0.5Si	HR	20Ti-10Cr	HR
20Ti-10Cb-2.0Si	HR	20Ti-20Cr	d
20Ti-20Cb-0.5Si	HR	20Ti-10Cb-15Cr	HR[b]
20Ti-20Cb-1.0Si	HR	20Ti-10Cb-10Al	HR[b]
20Ti-20Cb-2.0Si	HR	5Ti-20Cb-2.5Cr	HR[b]

a. HR = hot rolled, annealed, cold rolled.
   CR = cold rolled, annealed, cold rolled.
b. Did not fabricate.
c. Made using 99.95 purity vanadium - Bureau of Mines electrolytic process.
d. One of three ingots fabricated by hot rolling.

C. Tensile Results

1. Impurity Variables

Tensile data obtained under this program are reported in Table III for recrystallized 0.050 inch sheet specimens; yield strength values represent 0.2% offset. Elevated-temperature measurements were performed under a helium atmosphere (refer to Section II-D, above). Strength values for a few of the compositions listed in Table III had been reported during the previous alloy development programs. These earlier materials were prepared from melting stock of lower purity than that used during the current year. In addition, some of the earlier compositions required hot-working with the possible pickup of additional interstitials. As a consequence, alloys duplicated under the present program exhibit lower strength at elevated temperatures than similar but less pure compositions reported previously. One lot of vanadium was used during the current year, but two lots of columbium of different purity were used. Therefore, the tensile results reported in Table III reflect variations in raw material purity and fabricating techniques. It has also been shown that tensile properties obtained under the dynamic helium atmosphere are somewhat higher than values measured under a high vacuum, due to pickup of small quantities of interstitials during testing in the less pure atmosphere. The effects of impurities on mechanical properties are discussed in greater detail under Section G of this report. Many of the alloys in Table III were prepared using the same melting stock and fabricating procedures, enabling fairly accurate comparisons of alloying behavior to be made. In cases where material purity or processing variables were encountered, comparisons are less valid

2. V-20Cb Base Alloys

Compositions containing 20w/o columbium were studied at various titanium levels; some of these V-20Cb-Ti alloys also contained hafnium or zirconium. After earlier studies had indicated the V-5Ti-20Cb alloy to be the most promising at the 20w/o columbium level, a large number of small quaternary additions were made to the V-5Ti-20Cb base.

TABLE III

TENSILE PROPERTIES OF VANADIUM-BASE ALLOYS

Composition (w/o)	Room Temperature			2000° F			2200° F		
	Ultimate Tensile Strength (1000 psi)	Yield Strength (1000 psi)	Elong. (%)	Ultimate Tensile Strength (1000 psi)	Yield Strength (1000 psi)	Elong. (%)	Ultimate Tensile Strength (1000 psi)	Yield Strength (1000 psi)	Elong. (%)
20Cb	107.0	105.5	6	35.4	32.1	85			
0.2Ti-20Cb	113.6	100.8	23	32.1	29.3	92			
2.5Ti-20Cb	97.5	87.2	16	32.3	27.3	92			
2.5Ti-20Cb-0.25Hf				36.0	34.7	67			
2.5Ti-20Cb-0.5Hf				40.7	37.3	45	20.5	17.8	160
2.5Ti-20Cb-0.25Zr	94.6	84.8	13	34.3	33.1	36	19.7	17.6	123
2.5Ti-20Cb-0.5Zr	98.5	84.7	17	35.6	34.7	50			
5Ti-20Cb	110.4	95.0	16	37.4	34.0	73			
5Ti-20Cb-2.25Hf	102.3	84.5	23	36.7	33.4	57			
5Ti-20Cb-0.5Hf				43.2	41.7	45			
5Ti-20Cb-0.25Zr	105.5	91.0	24	34.0	29.9	101			
5Ti-20Cb-0.5Zr				39.7	36.7	52			
5Ti-20Cb-0.03C	108.5	92.3	15	38.7	36.2	27			
5Ti-20Cb-0.05C	107.0	96.6	15	43.9	41.2				
5Ti-20Cb-0.1C	120.0	101.1	20	40.7	37.7	35			
5Ti-20Cb-0.025B	107.0	[illegible]	8	43.9	41.1	9			

TABLE III (continued)

Composition (w/o)	Room Temperature Ultimate Tensile Strength (1000 psi)	Room Temperature Yield Strength (1000 psi)	Room Temperature Elong. (%)	2000° F Ultimate Tensile Strength (1000 psi)	2000° F Yield Strength (1000 psi)	2000° F Elong. (%)	2200° F Ultimate Tensile Strength (1000 psi)	2200° F Yield Strength (1000 psi)	2200° F Elong. (%)
5Ti-20Cb-0.05B	102.0	88.3	8	40.0	36.1	11			
5Ti-20Cb-0.1B	107.5	92.7	16	42.8	38.1	11			
5Ti-20Cb-0.025N	110.0	96.8	25				18.0	17.1	90
5Ti-20Cb-0.025O	107.7	101.2	24				19.1	17.6	65
5Ti-20Cb-0.05O	113.0	104.9	22				18.9	17.5	126
5Ti-20Cb-0.025C-0.025B	106.6	92.5	27				16.1	14.7	190
5Ti-20Cb-0.5Zr-0.03C	113.9	93.0	15	45.4	40.3	12	23.1	18.1	63
5Ti-25Cb	112.0	98.6	6	43.6	39.7	17			
30Cb	110.9	92.4	25	44.3	40.2	69			
5Ti-20Ta							23.5	17.5	87
5Ti-40Cb-0.03C	131.2	114.1	21	37.8	33.4	34			
4Hf-40Cb-0.1C	112.0	108.7	4	52.5	48.4				
60Cb	147.5	130.1	23	57.3	52.4	86	33.1	28.1	140
0.5Ti-60Cb	152.0		21	60.9	54.5	109	35.3	29.7	140
1.0Ti-60Cb	151.0	129.7	23	59.1	53.4	87	37.8	32.3	114
2.5Ti-60Cb	152.3	132.4	28	56.7	47.4	140			
0.5Hf-60Cb	149.9	130.1	21				37.9	33.2	65
1.0Hf-60Cb	149.6	131.5	19				44.4	40.6	37

TABLE III (continued)

Composition (w/o)	Room Temperature Ultimate Tensile Strength (1000 psi)	Room Temperature Yield Strength (1000 psi)	Room Temperature Elong. (%)	2000° F Ultimate Tensile Strength (1000 psi)	2000° F Yield Strength (1000 psi)	2000° F Elong. (%)	2200° F Ultimate Tensile Strength (1000 psi)	2200° F Yield Strength (1000 psi)	2200° F Elong. (%)
2.0Hf-60Cb	146.7	136.2	8				34.4	32.2	41
0.5Zr-60Cb	148.0	137.2	11				35.7	32.3	65
1.0Zr-60Cb	149.6	138.3	15				37.3	33.7	48
2.0Zr-60Cb							37.4	34.1	37
1Ti-60Cb-3Mo	129.0						37.0	34.2	21
1Ti-60Cb-5W	145.5	141.5	6				41.4	35.5	12
1Ti-60Cb-10Ta	153.0	142.0	17				37.4	35.1	82
1Hf-60Cb-5W				56.3	54.0	2			
1Ti-60Cb-0.025C	144.3	136.5	8				37.8	35.2	69
1Ti-60Cb-0.05C	161.9	152.0	13				35.2	33.4	55
1Ti-60Cb-0.025B	135.1	131.1	8				35.6	32.2	33
1Ti-60Cb-0.05B	152.0	141.0	8				35.6	32.6	63
1Ti-60Cb-0.025C-0.025B	153.7	147.6	10				35.1	31.3	30
1Ti-60Cb-0.025N	146.0	140.0	7				31.0	28.3	26
1Ti-60Cb-0.025O	147.3	135.5	12				33.7	31.2	22
1Ti-60Cb-0.05O	141.0	137.0	6				34.9	30.1	20
1Hf-60Cb-0.025O	185.0	183.0	8	55.4	46.2	33			
1Hf-60Cb-0.025C	170.8	158.9	6	59.4	51.9	14			
1Hf-60Cb-5W	179.3	175.0	8	56.3	54.0	2			
85Cb				52.5	46.8	35			
90Cb				43.7	40.6	50			

At room temperature, most of the V-20Cb base compositions had ultimate tensile strength values in the 100,000 to 110,000 psi range, with yield strengths of 90,000 to 100,000 psi. Strength levels fell to the 35,000 to 45,000 psi range at 2000°F, and to about 20,000 psi at 2200°F.

Solid-solution metallic additives to V-2.5Ti-20Cb included hafnium or zirconium up to 0.5w/o each; the 0.5w/o hafnium additive produced the greatest increase in 2000°F tensile strength. This quantity of hafnium also improved the strength of the V-5Ti-20Cb base at 2000°F (the value for V-5Ti-20Cb reported in Table III was obtained on material using the more recent, higher-purity vanadium and columbium). Small additions (0.025 to 0.1w/o) of boron and carbon produced a moderate improvement in the short-time 2000°F strength properties of the V-5Ti-20Cb base. Oxygen (0.025 and 0.05w/o) and nitrogen (0.025w/o) were added to the V-5Ti-20Cb base and tensile tested at 2200°F; ultimate tensile strength values were 18,000 to 19,000 psi. A V-5Ti-20Cb-0.5Zr-0.03C alloy had a strength of 23,100 psi at this temperature.

3. <u>V-60Cb Base Alloys</u>

Substantially increased strength properties at temperatures up to 2200°F without a reduction in fabricability were noted for alloys based on V-60Cb. At room temperature, ultimate tensile strength values were mostly above 145,000 psi with one composition as high as 180,000 psi. At 2000°F, ultimate strengths were in the 53,000 to 61,000 psi range, with yield strength values above 47,000 psi. The V-60Cb base materials were also relatively strong at 2200°F where ultimate tensile strength values of 33,000 to over 44,000 psi and yield strengths ranging from 28,000 to 40,600 psi were measured. These results represent a marked improvement over V-5Ti-20Cb alloys, especially at the higher temperatures. At 2000°F the V-0.5Ti-60Cb alloy had an ultimate tensile strength of 60,900 psi using the new higher-purity raw materials. Titanium, hafnium, and zirconium were added to the V-60Cb base at levels up to 2 or 2.5w/o, and tensile tests show that 1w/o hafnium produced the highest 2200°F strength of any alloy studied to date (44,400 psi). The effects of these additions on elevated-temperature strength are illustrated in Figure 1. Complexing of the V-1Ti-60Cb base with 5w/o tungsten produced a moderate increase in

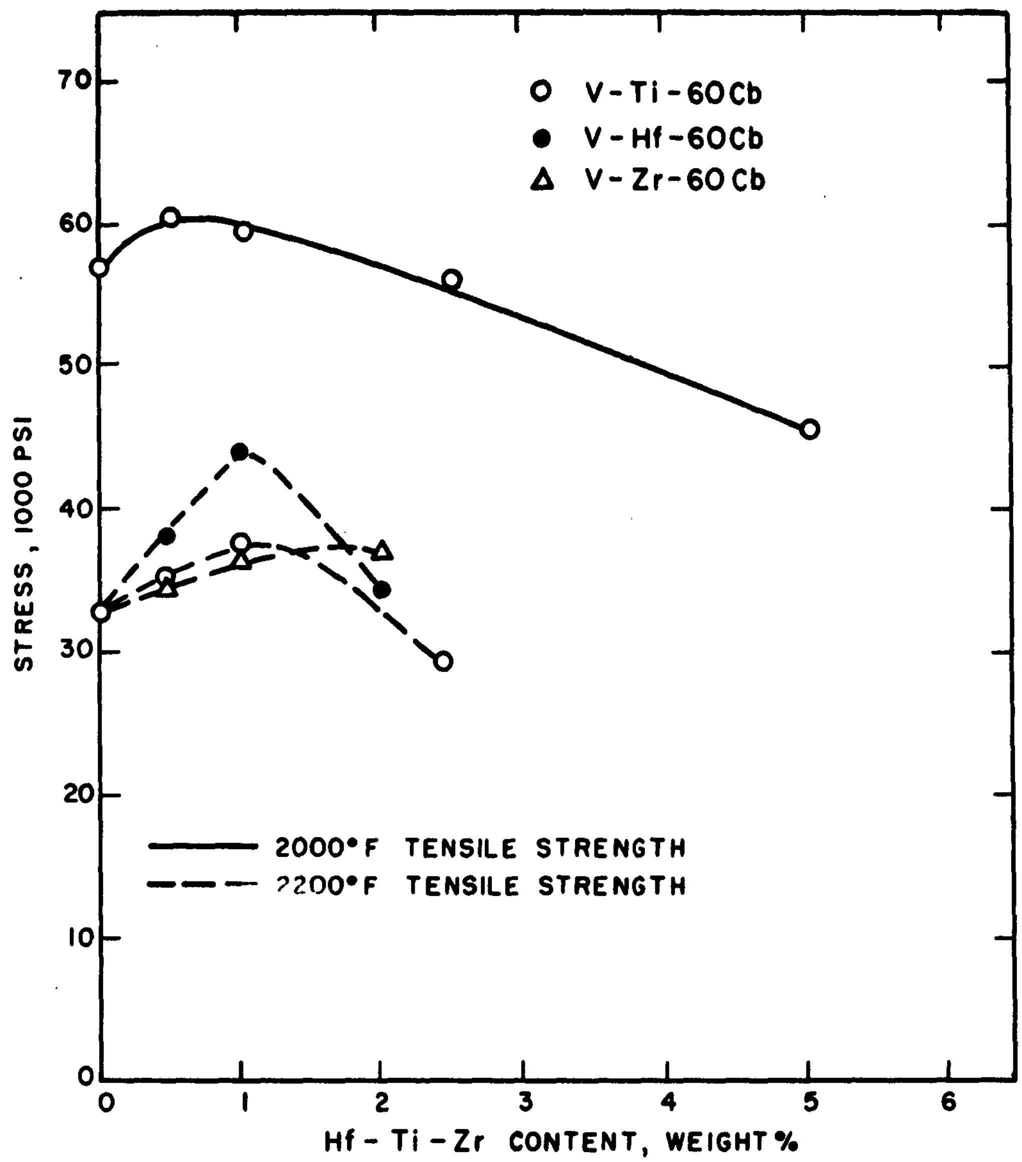


FIG. 1 - INFLUENCE OF TITANIUM, HAFNIUM, OR ZIRCONIUM UPON THE ELEVATED TEMPERATURE STRENGTH OF V-60Cb.

2200°F strength, whereas the addition of 3w/o molybdenum or 10w/o tantalum did not improve the elevated-temperature strength of the V-1Ti-60Cb base. Additions of 0.025 and 0.05w/o boron, carbon, nitrogen, and oxygen were made to V-1Ti-60Cb. In general, the short-time tensile properties were not significantly improved, although stress-rupture life (discussed in a subsequent section) was markedly increased by carbon, nitrogen, and oxygen.

4. Miscellaneous Alloys

In addition to the data for compositions based on V-20Cb and V-60Cb, Table III includes test results for other V-Cb binary alloys and several ternary and more complex alloys containing 25 to 40w/o columbium. During the previous year, attempts to fabricate the V-30Cb alloy were unsuccessful. Recently prepared material, using very high purity melting stock, fabricated readily to sheet and had a higher 2000°F strength than V-20Cb or V-40Cb (refer to Figure 6 of ARF 2191-6, Final Report under Contract NOas 60-6056-c). The columbium-rich end of the V-Cb system was screened by 2000°F tensile tests. These data show that the strength values decrease as the columbium levels exceed the 60 to 75w/o Cb range.

Two compositions containing 40w/o columbium were evaluated. The V-4Hf-40Cb-0.1C alloy had an ultimate strength of 52,500 psi at 2000°F, but this material had only 4% tensile elongation at room temperature. Much lower 2000°F strength (37,800 psi) was reported for a V-5Ti-40Cb-0.03C composition. Data for a V-5Ti-20Ta alloy show that the 2000°F tensile strength was reduced by replacing columbium with tantalum.

D. Stress-Rupture Properties

Alloys exhibiting the highest elevated-temperature tensile strength values were stress-rupture tested at 2000°F. Table IV summarizes the results of these tests, and data for V-5Ti-20Cb and V-5Ti-20Cb-0.25C obtained during the previous year are included for purposes of comparison.

Additions of carbon and/or boron to the V-5Ti-20Cb base produced improved long-time strength properties at 2000°F. The maximum increase was obtained at the 0.05w/o carbon level. Boron, carbon, oxygen, and nitrogen up to 0.05w/o were added to a V-1Ti-60Cb base. The most

TABLE IV

2000°F STRESS RUPTURE PROPERTIES OF VANADIUM-BASE ALLOYS

Composition (w/o)	Stress (1000 psi)	Fracture Time (hr)	Elong. (%)
5Ti-20Cb	8.5	4.1	20
5Ti-20Cb-0.03C	10.0	18.0	4
5Ti-20Cb-0.05C	10.0	22.0	38
5Ti-20Cb-0.1C	10.0	9.7	56
5Ti-20Cb-0.25C	8.0	21.7	64
5Ti-20Cb-0.025B	10.0	9.0	65
5Ti-20Cb-0.05B	10.0	8.5	50
5Ti-20Cb-0.025C-0.025B	10.0	3.4	83
1Ti-60Cb	10.0	13.4	36
1Ti-60Cb-0.025C	10.0	25.0	4
1Ti-60Cb-0.05C	10.0	27.3	34
1Ti-60Cb-0.05O	10.0	24.0	25
1Ti-60Cb-0.025N	10.0	19.0	6
1Ti-60Cb-0.05N	10.0	20.5	9
1Ti-60Cb-0.025B	10.0	16.0	27
1Ti-60Cb-0.05B	10.0	15.0	21
1Ti-60Cb-3Mo	10.0	9.0	x
1Ti-60Cb-5W	10.0	13.4	x
1Ti-60Cb-10Ta	10.0	47.7	24
1Hf-60Cb	10.0	27.4	23
1Hf-60Cb-5W	10.0	14.4	7

x Brittle fracture - No elongation.

effective quaternary additions were 0.05w/o carbon and oxygen, although both 0.025 and 0.05w/o nitrogen also improved the stress-rupture life of the base alloy. Boron additions produced very little changes in the long-time strength properties of V-1Ti-60Cb. Molybdenum (3w/o) or tungsten (5w/o) added to V-1Ti-60Cb did not improve the 2000°F stress-rupture life and brittle failures occurred. Microexamination of the stressed specimens revealed the presence of discontinuous grain-boundary precipitates. Subsequent evaluations of the alloys containing molybdenum or tungsten showed that room-temperature embrittlement was produced by stressing at 5000 psi at 2000°F in seven hours. The addition of 10w/o tantalum to the V-1Ti-60Cb base had the best long-time strength of all compositions under study; failure occurred in 47.7 hours under a 10,000 psi load at 2000°F. When the V-60Cb base was complexed with 1w/o hafnium, the stress-rupture life was greatly improved compared to that of V-1Ti-60Cb. This increase substantiates the tensile data which indicated that hafnium is a more potent strengther than titanium.

E. Alloys for 1200°F Service

Most of the solid-solution and dispersion-strengthened alloys described in the preceding sections are intended for service at 2000°F or above. A second category of vanadium-base alloys involves applications at or near 1200°F where previous studies indicated some promise for age-hardenable V-Ti-(Cb) base alloys containing silicon. Solid-solution V-Ti-(Cb) base materials containing chromium were also evaluated for 1200°F use.

1. Age-Hardening Alloys

Silicon at the 0.5, 1, and 2w/o levels was added to V-20Ti base compositions which also contained up to 40w/o columbium. In addition, a V-10Ti-20Cb-0.5Si alloy was studied. These alloys were annealed for 1/2 hour at 2400°F, water-quenched, then aged at 1200°F for times ranging from 0.1 to 100 hours. Aging curves for these alloys, based on Vickers 10 kg hardness measurements, are shown in Figures 2 through 5.

The effects of 0.5, 1, and 2w/o silicon on the aging characteristics of V-20Ti are illustrated in Figure 2. Virtually no age hardening was exhibited at the 0.5w/o silicon level. At 1w/o silicon, the hardness curve appears to be rising gradually at 100 hours; no precipitates were visible in the microstructures of the V-20Ti-1Si specimens aged at 1200° F up to 100 hours. Increasing the silicon content to 2w/o produced excessive hardening and overaging in less than 100 hours, and grain-boundary silicides were observed after the 100-hour treatment at 1200° F.

Figure 3 illustrates the aging response of the V-20Ti-20Cb base composition with 0.5 and 1w/o silicon. The alloy containing 0.5w/o silicon exhibited only a slight hardness increase and begins to soften after 10 hours. Silicon at the 1w/o level produced excessive hardening with resultant room-temperature embrittlement after 24 hours at 1200°F. Reducing the titanium content from 20 to 10w/o while maintaining 20w/o columbium and 0.5w/o silicon results in an alloy which exhibits potential for 1200° F service, as shown in Figure 4. Aging curves in Figure 4 also include V-20Ti-0.5Si alloys containing 20 and 40w/o columbium. The curve for V-20Ti-40Cb-0.5Si indicates promise, as overaging is not observed after 100 hours, whereas a hardness peak for V-20Ti-20Cb-0.5Si is reached in about 10 hours at 1200° F. Silicides observed in the alloy containing 40w/o columbium appear to be agglomerating after the 100-hour aging treatment; this effect was observed mostly within the grains and not at the grain boundaries.

Studies of the V-Ti-O system indicated that some compositions at the 0.2w/o oxygen level were age-hardenable in the 1000° to 1400°F range.[1] Although alloys currently under investigation contain lower amounts of oxygen, age-hardening studies were conducted on the V-5Ti-20Cb and V-1Ti-60Cb base compositions having oxygen additions of 0.05w/o. Based on analyses of the raw materials, the total oxygen levels are probably slightly above 0.1w/o. Aging curves for these compositions are shown in Figure 5. Overaging occurs when the V-5Ti-20Cb-0.05O alloy is heat treated for 100 hours at 1200° F, and small amounts of a fine, rounded

(1) S. A. Komjathy, R. H. Read, and W. Rostoker, WADD Technical Report 59-483, July, 1959.

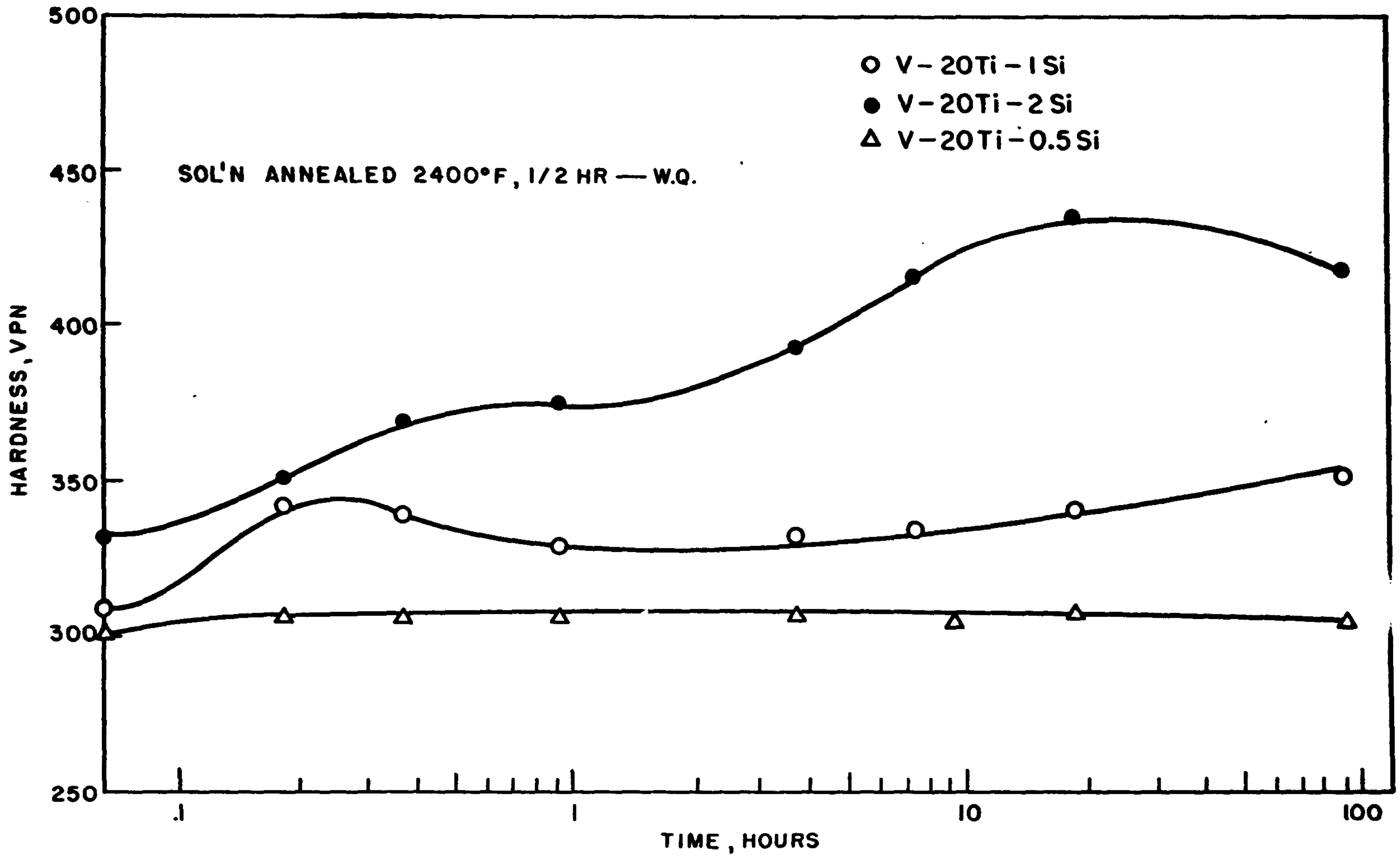


FIG. 2 - AGING RESPONSE AT 1200°F FOR V-20Ti-Si ALLOYS

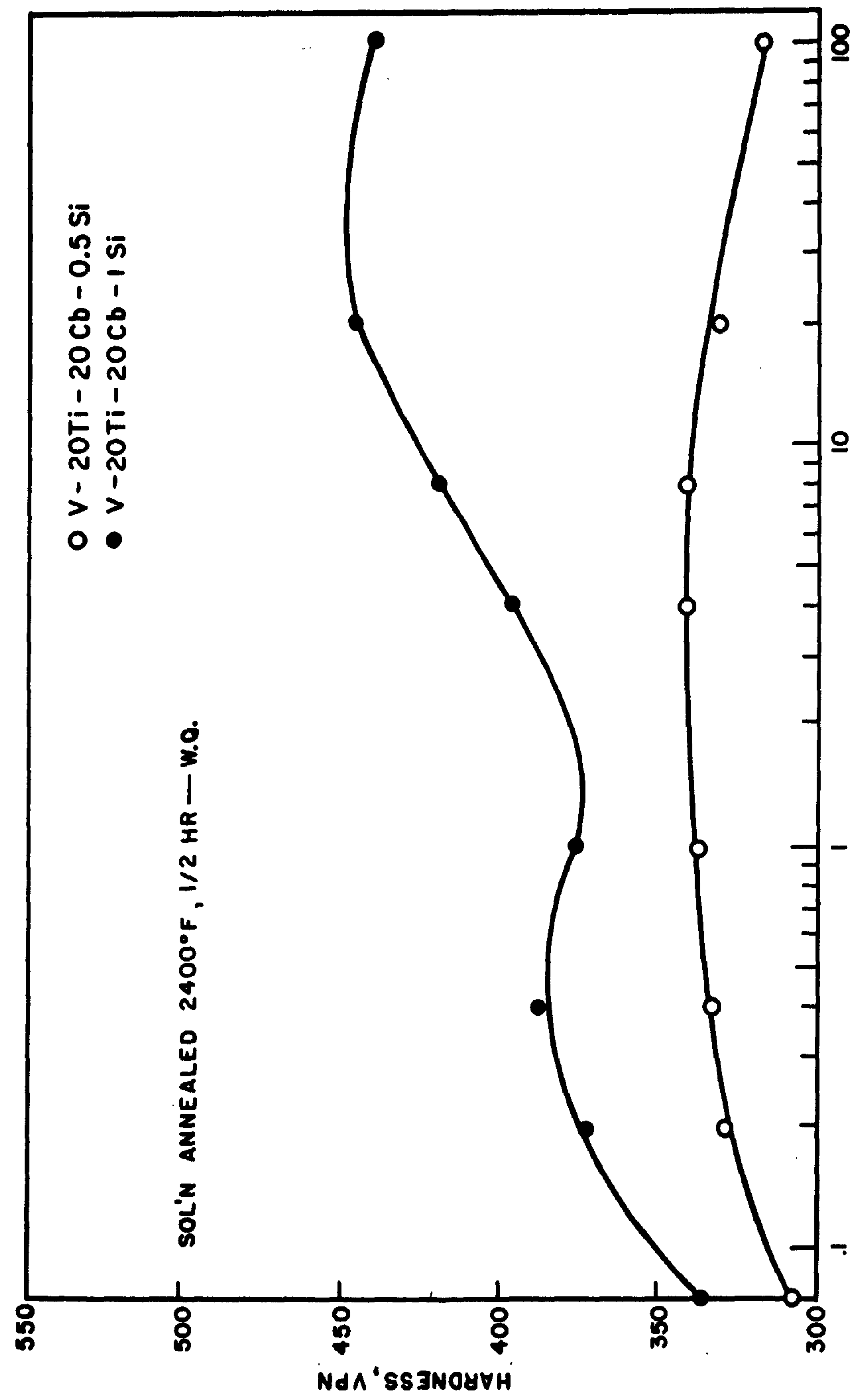


FIG. 3 - AGING RESPONSE AT 1200°F FOR V-20Ti-20Cb-Si ALLOYS

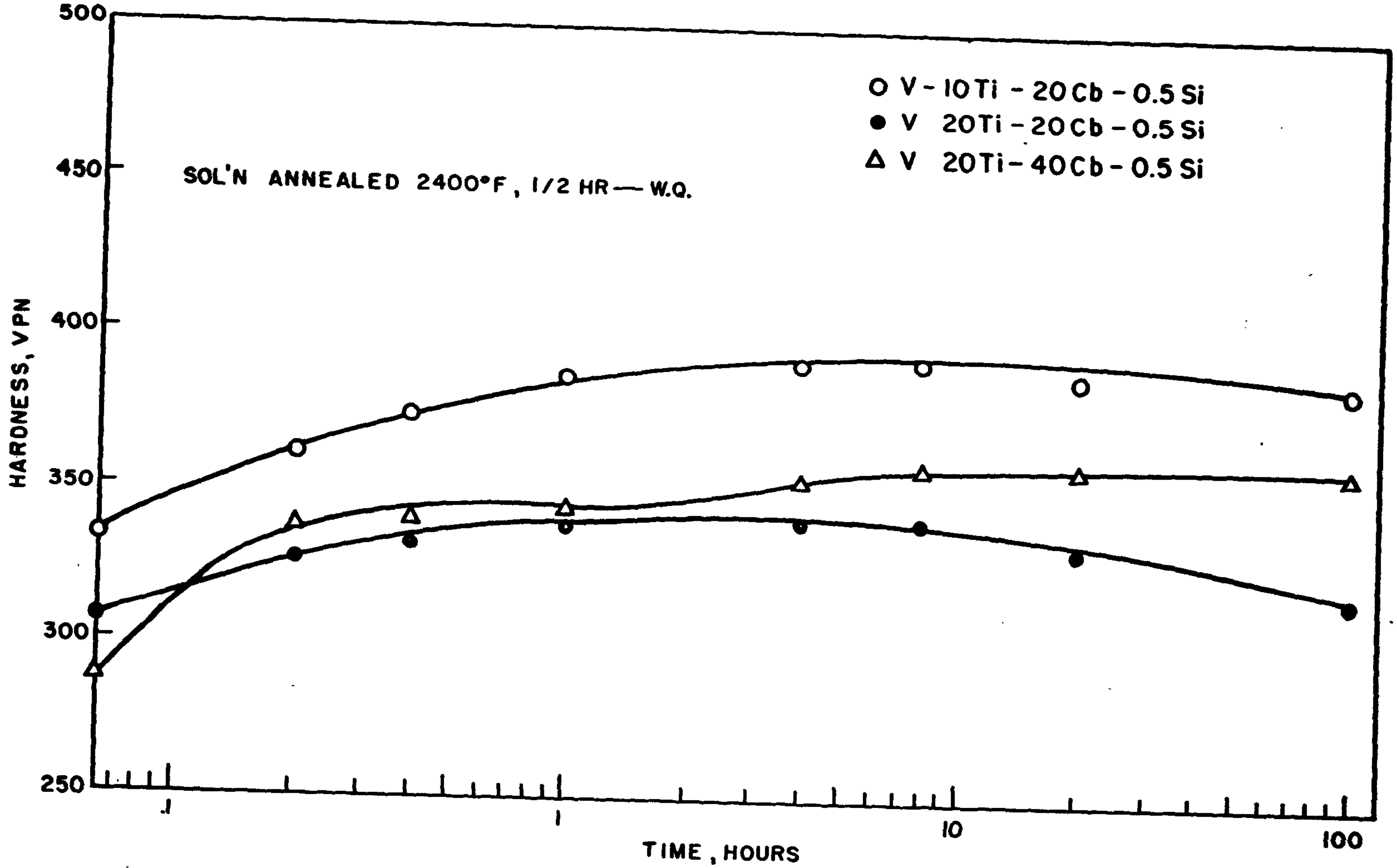


FIG. 4 - AGING RESPONSE AT 1200°F FOR V-Ti-Cb-0.5Si ALLOYS

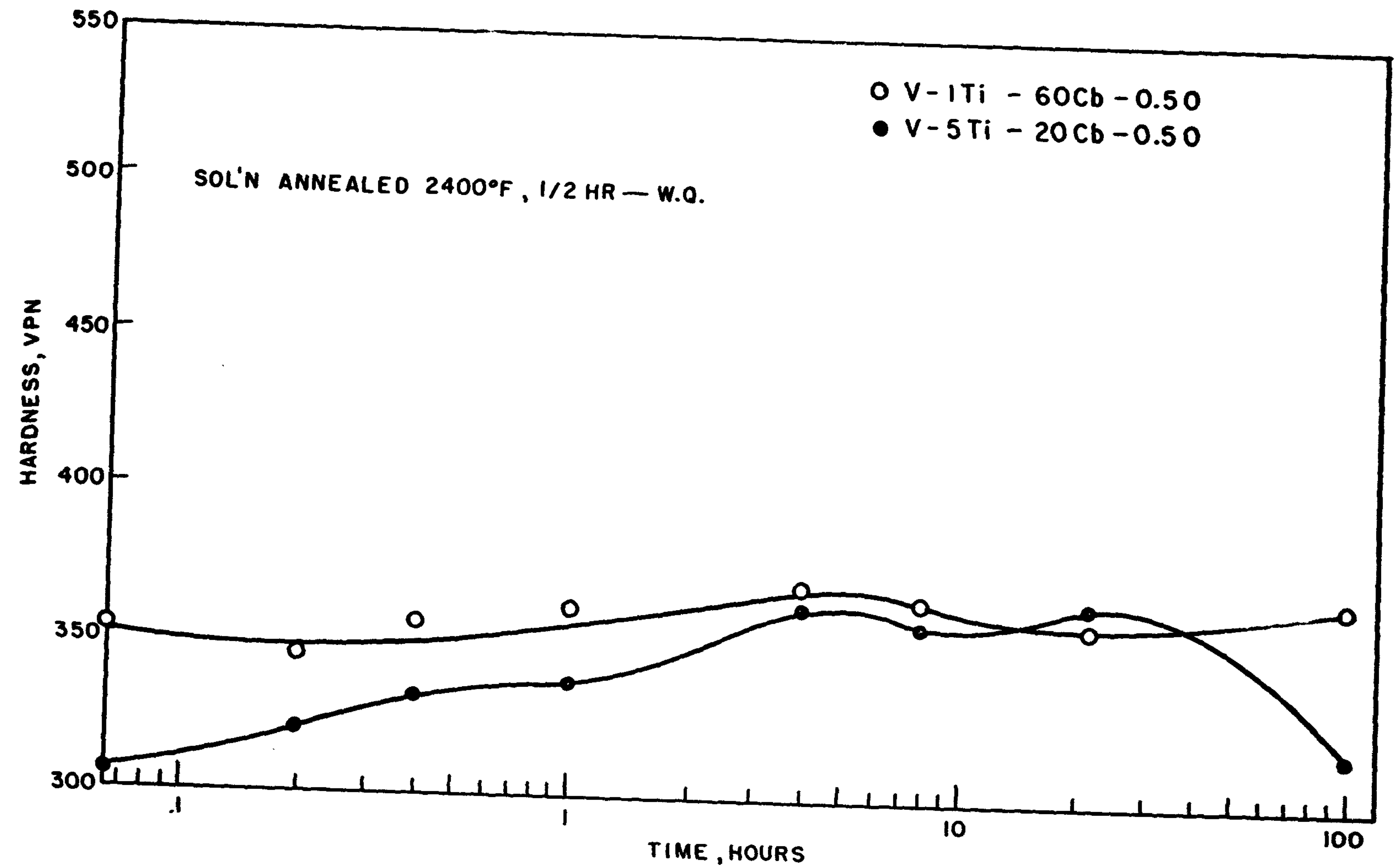


FIG. 5 - AGING RESPONSE AT 1200° F FOR V-Ti-Cb-O ALLOYS

grain-boundary precipitate are noted in the microstructures. Hardness levels of the V-1Ti-60Cb-0.05O compositions are increasing slightly after aging for 100 hours, although the hardness increase amounts to only 30 VPN above the hardness of the solution-annealed material. No microstructural changes were observed in the latter alloy after the 100-hour thermal treatment. It is possible that slightly higher oxygen levels would increase the age-hardening effects.

Tensile, stress-rupture, and creep data were not obtained on the V-Ti-(Cb)-Si or on the V-Ti-Cb-O compositions because the major alloy development efforts were concentrated on materials for higher-temperature use (2000°F or above). Previous studies of V-5Ti-20Cb-1Si showed 0.1% deformation after 100 hours at 1200°F under a stress of 50,000 psi, but the specimen became embrittled during the exposure. A lower silicon alloy, V-5Ti-20Cb-0.5Si exhibited 1% plastic deformation under the same conditions, and no embrittlement was observed. To be competitive with currently available materials, the V-5Ti-20Cb base would have to withstand 64,000 psi for 100 hours at 1200°F with 0.1% plastic deformation.

2. Solid-Solution Alloys

Earlier vanadium alloy studies indicated that V-Ti-Cr compositions exhibited promise for 1200°F applications. Recent efforts were devoted to four alloys containing chromium in amounts ranging from 10 to 25w/o; additionally, an ingot of V-20Ti-10Cb-10Al was melted. These compositions are listed in Table II. Only the V-20Ti-10Cr material fabricated satisfactorily, although one of three ingots of V-20Ti-20Cr was also rendered to sheet. The remainder of the ingots cracked severely during hot working.

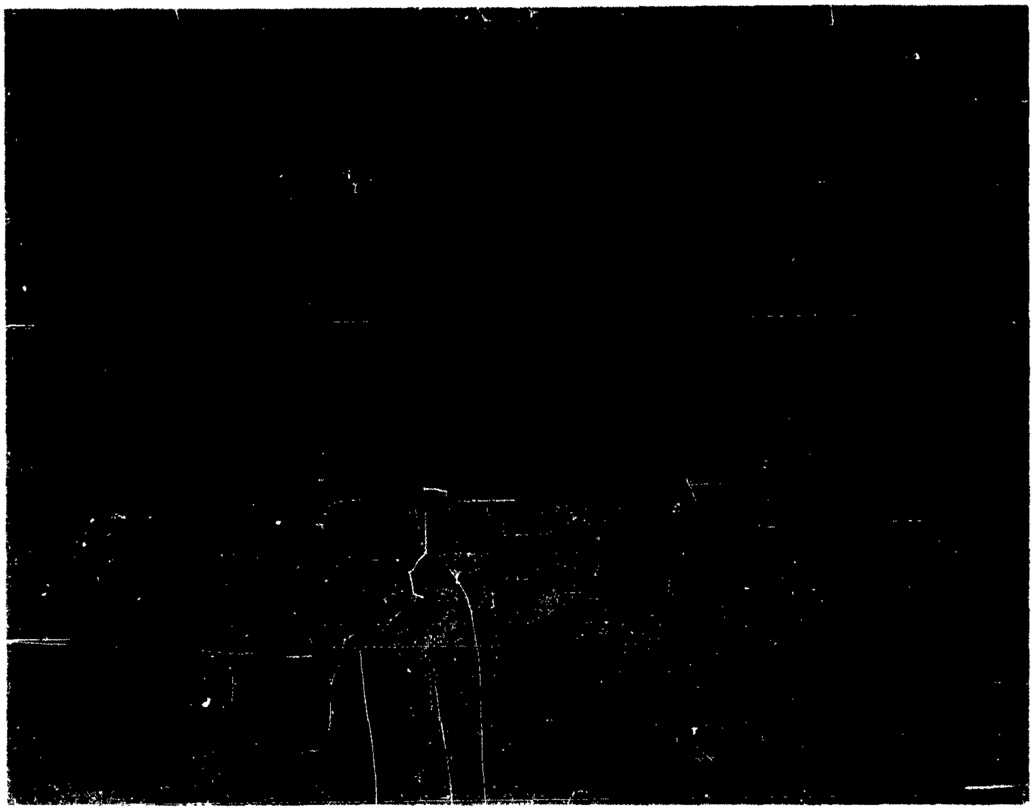
Tensile results for the V-20Ti-10Cr material show an ultimate strength of 78,400 psi at 1200°F, whereas the V-20Ti-20Cr alloy had a strength of 86,700 psi at this temperature. When stressed at 60,000 psi at 1200°F, the V-20Ti-10Cr specimen failed in 4 hours. Specimens of the higher-chromium composition were not available for stress-rupture or creep testing because of the inability to fabricate additional ingots.

F. Weldability

Sheet specimens of V-5Ti-20Cb and V-60Cb alloys were welded and subsequently evaluated by bend and tensile tests. Fully annealed 0.050 inch sheet samples were butt welded by tungsten-inert gas shielding techniques. The inert gas was allowed to flow on both sides of the welded sheet; trailing gas shielding was also employed. Figure 6 illustrates welds in the two alloys and a welded joint between V-5Ti-20Cb and unalloyed columbium.

After welding, the sheets were machined into tensile and bend test specimens; no post welding heat treatments were given. Tensile and bend tests showed that the room-temperature properties of the parent metal were retained. As-welded V-5Ti-20Cb passed a 1.2t bend, the same value obtained for the parent metal. Similar results were obtained for V-60Cb where both the parent material and the welded sheet passed a 2.5t bend. Room-temperature tensile test results for the as-welded specimens approximated the values obtained on the unwelded stock. A slight reduction of 2000°F tensile properties was observed in the welded materials: ultimate strength values were reduced by about 4000 to 5000 psi, and a smaller loss of yield strength (2000 to 3000 psi) occurred. In addition, the welded specimens exhibited a significant loss of tensile elongation at 2000°F, where values of 8 and 9% were measured for V-5Ti-20Cb and V-60Cb, respectively. This reduced elevated-temperature ductility was attributed in part to the significantly increased grain size and possibly to the pickup of interstitial impurities during welding. Annealing both alloys (unwelded) at 2800°F produced a coarse-grained structure which also exhibited low tensile elongations (6%) when tested at 2000°F.

Specimens of V-5Ti-20Cb and V-60Cb were also welded to unalloyed columbium. Tensile data were not obtained since the relatively low strength of the columbium sheet would cause failure to occur in this material. However, bend tests were conducted at room temperature where the weldment of V-5Ti-20Cb to columbium passed a 2.5t bend, and V-60Cb to columbium passed a 5t bend. Failures during more severe bends always occurred on the columbium-rich side.



Neg. No. 21092 Full Scale

Figure 6

Welded specimens of V-Cb sheet alloys. From left to right the materials are: (1) V-5Ti-20Cb--unalloyed columbium, (2) V-5Ti-20Cb, and (3) V-60Cb. Test specimens bent in the weld (shown below the respective sheets) passed 2.5t, 1.2t, and 2.5t bends.

G. Interstitial Impurity Effects

During the course of these vanadium alloy studies, the pronounced effect of intentional additions of carbon, nitrogen, and oxygen on mechanical properties have been demonstrated by elevated-temperature tensile and stress-rupture tests. The 200-gram ingots used in these investigations were prepared by nonconsumable-electrode arc-melting under an atmosphere of argon, and chemical analyses indicated that intentional additions of carbon and oxygen were retained during melting. These analyses, however, showed slightly higher interstitial levels than were indicated by the analyses of the raw materials, suggesting that traces of oxygen (and possibly nitrogen) are picked up during melting. It has also been observed that the increasing purity of melting stock used for these alloy development programs has resulted in improved fabricability and, in general, somewhat lower elevated-temperature tensile strengths. With the improved fabricability, many compositions, which under previous programs could be rendered to sheet only by hot working, could more recently be cold-rolled using the higher-purity raw materials. Hot working, although carried out in evacuated stainless steel cans, undoubtedly resulted in the pickup of very small quantities of interstitials. It has further been demonstrated that additional interstitials were picked up during the elevated-temperature tensile tests conducted under a dynamic helium atmosphere. The test data reported during this program and the previous programs represent materials whose initial interstitial levels have been altered to some extent during melting, hot working, and elevated-temperature testing, as discussed below:

1. Effects of Raw Material Purity

During the three years of alloy development, the impurity levels of the vanadium melting stock have been reduced as shown by the following vendor's analyses (w/o):

Year	C	O	N	H
1959	0.043	0.09	0.037	0.007
1960	0.032	0.062	0.025	0.003
1961	0.024	0.055	0.039	0.0018

Columbium analyses have also varied, although the most recent material (electron-beam melted) has a higher impurity content than earlier stock. The vendor's analyses show the extent of variations in interstitial levels(w/o):

Year	C	O	N	H
1959	0.0021	0.0325	0.0167	< 1 ppm
1960-61	0.003	0.0135	0.0098	not reported
1961	0.008	0.04	0.01	0.0035

It has been observed that V-5Ti-20Cb and other alloys prepared from the more recent melting stock can be cold rolled, whereas similar compositions made with earlier materials required hot-working for ingot breakdown. This substantial improvement in fabricability in the case of V-5Ti-20Cb was achieved by a reduction of total interstitial content (carbon, oxygen, and nitrogen) from about 0.136w/o for the 1959 material to approximately 0.106w/o for the most recent melting stock. It is more difficult to assess the effect of raw material purity on elevated-temperature properties because most of the ingots using the lower-purity stock required hot-working, and further pickup of interstitials during working may have affected tensile test results. In general, however, a downward trend in elevated-temperature tensile strength values has been observed as raw material purity increased. The particular interstitial most responsible for property changes has not been determined.

2. Impurity Pickup During Processing

Analyses conducted on 200-gram ingots prepared by nonconsumable-electrode arc-melting under an inert atmosphere have indicated that relatively small and variable amounts of carbon and oxygen were picked up during melting. Total carbon and oxygen levels after melting were about 0.023w/o higher than indicated by the raw material analysis. It is believed that these results may reflect some experimental error, as the source of carbon contamination could not be established. However, it is possible that interstitials may be picked up, or even lost, during the melting operation. Tensile properties of a V-5Ti-20Cb material prepared by consumable-electrode arc-melting under vacuum were 1600 psi lower at 2000°F than for the same alloy using the identical melting stock but melted under inert atmosphere.

Sheet stock of V-5Ti-20Cb prepared by different melting techniques and working procedures was analyzed for carbon, oxygen, and nitrogen. Prior to melting, the total interstitial contents ranged from 0. 106 to 0. 145w/o; after melting and hot or cold rolling, the total impurities increased to a range of 0. 145 to 0. 179w/o. The major increases were found in carbon and oxygen levels. Thus, a small but possible significant rise in impurity levels is produced during melting and fabrication.

3. Impurity Pickup During Elevated-Temperature Testing

Tensile data for V-5Ti-20Cb at 2000° F show that sheet material tested under a high vacuum has an ultimate tensile strength lower by at least 6000 psi than similar stock tested in the dynamic helium atmosphere used to screen the alloys studied under these programs. This effect was also noted for V-1Ti-60Cb specimens. In all cases, tensile elongations on materials tested in the lower-purity atmosphere were greatly reduced compared to the values obtained under vacuum. It is apparent that some interstitials were picked up during the tests in the dynamic helium, and these impurities caused increased strength and reduced tensile elongation at elevated temperatures. These specimens were heated very rapidly to the test temperature and held for 10 minutes prior to application of the load. After testing, a very light discoloration was observed on the vanadium alloy surfaces. Microhardness surveys did not reveal hard surface layers, so it is possible that the impurities diffused throughout the 0. 050 inch sheets. Hardness levels after many 2000° or 2200° F tests were not substantially increased, and a large number of the specimens tested under dynamic helium could be severely bent at room temperature. The extent of impurity pickup during testing has not been determined, although this subject will be explored in greater detail under the pilot evaluation program in order to establish optimum interstitial levels for the selection of compositions for the large ingots.

H. Salt Corrosion Studies

Results of the mechanical tests conducted to determine the salt corrosion susceptibility of the V-5Ti-20Cb alloy are presented in Table V. The salt, containing seven parts of sodium chloride to one part of magnesium chloride, was applied by painting the specimens with a 10% solution and evaporating the water.

Table V shows that the V-5Ti-20Cb material exhibited virtually no susceptibility to salt corrosion at 800° F. Metallographic examination of both salted and bare samples revealed similar depth of oxide coatings (less than 0.001 inch), and neither specimen exhibited surface cracks. At 1000° F, slight evidence of salt corrosion susceptibility was observed although the tensile properties were not markedly affected. A somewhat heavier oxide layer (0.003 inch thick) formed on the specimen exposed to salt compared to the 0.001 inch thick layer on the bare specimen. Some light pitting was also noted on the surface of the salted sample although no cracks were observed on the 1000° F test specimens.

I. Miscellaneous Evaluations

Specimens of vanadium alloy sheet have been sent to a number of outside organizations for the evaluation of properties of special interest to these investigators. A list of these organizations and the type of studies performed is presented in Table VI. Information concerning test results has been submitted by five of the investigators. Additional data received after the conclusion of this program will be incorporated in reports issued under Contract NOw 62-0101-c The following brief discussions cover the data accumulated to date:

(1) Aerojet-General Nucleonics. V-5Ti-20Cb compares favorably with Haynes 25 alloy and type 316 stainless steel in resistance to mercury in the 1000° to 1200° F range.

(2) Naval Research Laboratory. Zinc coatings provide oxidation protection to V-5Ti-20Cb for more than 60 hours at 1600° F, but failed immediately at 1800° F. The V-60Cb alloy was protected by zinc for periods up to 1 hour at 1800° F.

(3) Argonne National Laboratory. The vanadium-base sheet materials supplied for welding studies exhibited excellent weldability and fabricability. After welding into tubular shape, swaging was used to reduce diameter and wall thickness. Electron-beam welded specimens were returned

TABLE V

SALT CORROSION TEST DATA FOR V-5Ti-20Cb ALLOY

Test Condition			Duration of Test (hrs)	Elong. (%)	Room Temperature Tensile Properties		
Temp. (°F)	Stress (1000 psi)	Atmos.[a]			Ultimate Tensile Strength (1000 psi)	Yield Strength (1000 psi)	Elong. (%)
800	55.0	Inert - u	112.0	0	113.3	97.6	17
800	75.0	Air - u	92.0	0	109.7	98.2	16
800	85.0	Air - u	97.0	2	111.2	101.4	15
800	85.0	Air - s	124.3	3	108.9	99.7	15
1000	55.0	Inert - u	92.0	0	112.7	101.7	16
1000	75.0	Air - u	92.0	2	108.9	102.2	15
1000	75.0	Air - s	137.5	2+	114.3	103.2	9[b]

a. u = unsalted s = salted
b. Broke outside gage mark.

TABLE VI

VANADIUM ALLOY STOCK SENT TO OTHER ORGANIZATIONS

Organization and Address	Sheet Composition (w/o)	Purpose Desired
National Aeronautics and Space Adminstration Lewis Research Center 21000 Brookpark Road Cleveland 35, Ohio	V-5Ti-20Cb	Test in static reflux capsule
Aerojet General Nucleonics San Ramon, California	V-10Ti, V-20Ti V-5Ti-20Cb	Compatibility with mercury
Naval Research Laboratory Washington 25, D. C.	V-5Ti-20Cb V-60Cb	Test zinc coatings for oxidation protection
Argonne National Laboratory Post Office Box 299 Argonne, Illinois	V-5Ti-20Cb V-10Ti-3Cb V-10Ti-3Ta	Weldability and compatibility with sodium
McDonnel Aircraft Corp. Post Office Box 516 St. Louis 66, Missouri	V-5Ti-20Cb V-1Ti-60Cb	Testing oxidation resistant coatings
Argonne National Laboratory 9700 South Cass Avenue Argonne, Illinois	V-Ti-(Cb, Mo Ta, W) Approx. 14 alloys	Sodium and platinum compatibility tests
Boeing Airplane Company Aero Space Division Post Office Box 3707 Seattle 24, Washington	V-5Ti-20Cb	Vacuum tensile testing at elevated temperatures
U.S. Department of the Interior Bureau of Mines College Park Research Center College Park, Maryland	V-(2.5-10)Ti-(20-60)Cb Approx. 10 alloys	Sodium corrosion studies

to the Foundation for examination; this sheet stock, welded at 130 kv and 3 milliamperes showed good penetration without excessive spatter.

(4) McDonnell Aircraft Corporation. Specimens of V-5Ti-20Cb and V-60Cb were slurry-coated with an aluminum alloy which was subsequently reacted with the vanadium base at 1900°F. Protection was afforded for 4 hours at 1800°F; at 2000°F, failures occurred in air in 2.25 and 1.5 hours for V-5Ti-20Cb and V-60Cb, respectively. Both alloys withstood exposure to 2500°F air for 15 minutes.

(5) Boeing Airplane Company. V-5Ti-20Cb was tensile tested under high vacuum at temperatures up to 2700°F, using a strain rate of 0.005 in/in/min to 0.6% offset.

The following results were obtained:

Temp. (°F)	Ultimate Tensile Strength(psi)	Yield Point (psi)	Elongation (% in 1 in.)
1810	38,600	26,800	29
2000	25,800	23,600	> 69
2500	5,540	4,280	> 37
2700	3,880	3,800	> 64

The same lot of sheet stock prepared with electron-beam melted columbium and tested at the Foundation had an ultimate tensile strength of 27,200 psi under vacuum and 33,800 psi under dynamic helium at 2000°F.

IV. SUMMARY AND CONCLUSIONS

The studies described herein are a continuation of vanadium alloy development efforts conducted under Contracts NOas 59-6050-c and NOas 60-6056-c. Alloys based on the vanadium-columbium system have exhibited outstanding fabricability and weldability with excellent retention of strength to at least 2200°F. During the current year, ternary and more complex alloys containing up to 60w/o columbium were investigated, and these studies conclude the major portion of the alloy development work.

Current related programs include the development of oxidation-protective coatings under Contract NOw 61-0806-c and a pilot evaluation of vanadium alloys under Contract NOw 62-0101-c. A relatively small but important area of alloy development--the optimizing of interstitial levels--will be investigated under the pilot evaluation program prior to selection of compositions for the large ingots.

Alloys based on V-Cb were prepared by nonconsumable-electrode arc-melting and evaluated at temperatures up to 2200°F. The most promising compositions to date were based on V-5Ti-20Cb and V-60Cb. Excellent workability was indicated by the fact that many of the arc-melted ingots could be cold-rolled directly to 0.050 inch sheet, a reduction in thickness of about 90%. Fabricability was reduced by increased carbon, oxygen, and nitrogen levels. Sheet stock welded by tungsten-inert gas shielding techniques retained their excellent room-temperature bend ductility, and only a very slight reduction in 2000°F strength was noted. Most cold-rolled alloys were fully recrystallized at 2000°F, although small carbon or boron additions produced increases of 200° and 400°F respectively.

At the 20w/o columbium level, 5w/o titanium was the optimum addition for elevated-temperature strengthening. Addition of 0.05w/o carbon to V-5Ti-20Cb resulted in greatly improved stress-rupture life at 2000°F. Hafnium at the 1w/o level was found to be the most potent strengthener for the V-60Cb base; at 2200°F, an ultimate tensile strength of 44,400 psi was measured for V-1Hf-60Cb. Good strength at 2000°F was noted in V-0.5Ti-60Cb (60,900 psi ultimate), and 0.05w/o carbon markedly improved the long-time 2000°F strength properties of this composition. In general, alloys based on V-60Cb were at least 30% stronger than those based on V-5Ti-20Cb at temperatures up to 2000°F. The strength advantage of V-60Cb base materials was even greater at 2200°F.

On a density-corrected basis, the more promising vanadium alloys compare favorably in short-time strength at 2000° and 2200°F with Cb-10Ti-10Mo and other moderate-strength columbium alloys. Although the strength to density ratios of the vanadium base materials are somewhat below those of Mo-0.5Ti-0.07Zr at these temperatures, their excellent fabricability and weldability is an important consideration. Promising results for oxidation-protective coatings on V-60Cb base alloys under a current program indicate that these materials should find application at 2000° to at least 2200°F.

Age-hardening characteristics of vanadium-base alloys were investigated for possible applications at or near 1200°F. Aging curves showed considerable hardness increases without serious embrittlement or overaging in several alloys containing 1w/o silicon. Small quantities of oxygen (0.05w/o) added to V-5Ti-20Cb and V-1Ti-60Cb produced moderate age hardening effects at 1200°F, although overaging occurred in less than 100 hours for the V-5Ti-20Cb-0.05O material.

The pronounced effect of interstitial impurities (carbon, nitrogen, and oxygen) on elevated temperature strength properties was demonstrated by fabricability studies and elevated-temperature tensile evaluations. Interstitials present in the raw materials, and also those picked up during melting, hot working, and elevated-temperature tensile testing, resulted in reduced workability and increased strength at high temperatures. The extent to which these impurity levels were altered during processing or testing, and the particular element or elements most responsible for the mechanical property changes have not been determined. These variables must be explored prior to designating the optimum interstitial contents for compositions to be studied in greater detail under the current pilot evaluation program.

V. LOGBOOKS AND CONTRIBUTING PERSONNEL

The data accumulated on this program are recorded in Foundation Logbooks C-10872, -10873, -11175, -11184, and -11514.

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The following personnel contributed to the work reported herein:

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Respectfully submitted,

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